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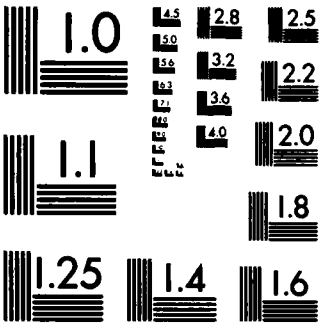

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Technical Memorandum

INVESTIGATION OF COHERENT ANTI-STOKES RAMAN SCATTERING MEASURMENTS IN CARBON ABLATION EXPERIMENTS

R. C. BENSON

H. Y. CHIU

THE JOHNS HOPKINS UNIVERSITY ■ APPLIED PHYSICS LABORATORY

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REPORT DOCUMENTATION PAGE

1. REPORT NUMBER: JHU/APL TG 1341

2. GOVT ACCESSION NO.

3. RECIPIENT'S CATALOG NUMBER

4. TITLE (*and Subtitle*): Investigation of Coherent Anti-Stokes Raman Scattering Measurements in Carbon Ablation Experiments

5. TYPE OF REPORT & PERIOD COVERED: Technical Memorandum

6. PERFORMING ORG. REPORT NUMBER: JHU/APL TG 1341

7. AUTHOR (*s*): R. C. Benson and H. Y. Chiu

8. CONTRACT OR GRANT NUMBER (*s*): N00024-83-C-5301

9. PERFORMING ORGANIZATION NAME & ADDRESS:
The Johns Hopkins University Applied Physics Laboratory
Johns Hopkins Road
Laurel, MD 20707

10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS: Task X8G1

11. CONTROLLING OFFICE NAME & ADDRESS:
The Johns Hopkins University Applied Physics Laboratory
Johns Hopkins Road
Laurel, MD 20707

12. REPORT DATE: June 1983

13. NUMBER OF PAGES: 18

14. MONITORING AGENCY NAME & ADDRESS:
The Johns Hopkins University Applied Physics Laboratory
Johns Hopkins Road
Laurel, MD 20707

15. SECURITY CLASS. (*of this report*): Unclassified

15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (*of this Report*): Approved for public release; distribution unlimited

17. DISTRIBUTION STATEMENT (*of the abstract entered in Block 20, if different from Report*)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (*Continue on reverse side if necessary and identify by block number*)

Raman scattering
graphite ablation
carbon monoxide

20. ABSTRACT (*Continue on reverse side if necessary and identify by block number*)

Investigations were made of the feasibility of using Raman techniques to measure the gaseous products of carbon oxidation. A coherent anti-Stokes Raman scattering system was assembled to measure carbon monoxide. Approximately 200 μm from the graphite surface, carbon monoxide concentrations in the range of 5 to 10% (40 to 80 Torr) were measured during the ablation of ATJ graphite in air at 2000 K. Recommendations were made for improvements to the experimental system.

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INVESTIGATION OF COHERENT ANTI-STOKES RAMAN SCATTERING MEASURMENTS IN CARBON ABLATION EXPERIMENTS

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ABSTRACT

Investigations were made of the feasibility of using Raman techniques to measure the gaseous products of carbon oxidation. A coherent anti-Stokes Raman scattering system was assembled to measure carbon monoxide. Approximately 200 μm from the graphite surface, carbon monoxide concentrations in the range of 5 to 10% (40 to 80 Torr) were measured during the ablation of ATJ graphite at 2000 K in air. Recommendations were made for improvements to the experimental system.

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ILLUSTRATIONS

1. INTRODUCTION

The combustion of solid carbon and other carbonaceous materials subjected to an oxidizing gas flow is of interest in understanding basic heterogeneous chemical processes and also in the application of carbon materials in aeronautical environments. The combustion rate of carbon depends on the chemical reactions occurring and also on the gas flow conditions. Both heterogeneous and homogeneous chemical reactions occur. However, there is some controversy regarding the relative importance of these reactions under various temperatures and flows.[1,2] Knowledge of the relationship between the chemistry and gas flow and the carbon oxidation is essential in order to develop an accurate theoretical model. At APL, this modeling is important to the Aeronautics Division because various components of high-speed missiles are made of carbon materials and are subjected to highly oxidizing gas flows. To achieve satisfactory engine performance, the amount of carbon ablation must not exceed prescribed limits.

Ideally, experiments are needed to determine the chemical aspects of carbon oxidation for various flow conditions. To our knowledge, no experiments have been conducted in which the concentration of chemical species has been measured as a function of distance from the carbon surface. The objective of this work was to determine the feasibility of using Raman techniques to obtain such measurements. Optical techniques offer the advantage that they are noninvasive, so that the flow field will not be perturbed during the measurement. However, the technique must be sensitive enough yet not be limited by the intense blackbody radiation from the hot carbon surface. Laser-induced fluorescence is quite sensitive, but it requires a tunable laser and is generally applicable only to atoms and diatomic molecules. Furthermore, collisional quenching and self-absorption must also be considered for quantitative measurements. Raman techniques are generally applicable to molecules and are unaffected by collisional processes or absorption corrections beause the incident photons are scattered, not absorbed. Spontaneous Raman scattering has several desirable features, the most important being that both the optical system and the interpretation of the data are quite simple. The relationship between the Raman signal and the species concentration and temperature is straightforward. The spatial resolution is excellent—approximately 20 μm for lenses of moderate focal length. Unfortunately, those advantages are often outweighed by low sensitivity and, especially in combustion applications, by background luminosity and laser-induced interferences.

The coherent anti-Stokes Raman scattering (CARS) technique, although much more complex than the spontaneous Raman method, overcomes most of the disadvantages of the latter when probing combustion processes. CARS is a rather strong process that results in signal levels many orders of magnitude larger than those from spontaneous Raman scattering. Because the CARS radiation is essentially a laser beam, all of it can be collected. On the other hand, in spontaneous Raman scattering the photons are scattered over 4π steradians and are collected only over a limited solid angle. An important consequence of the coherent nature of the CARS radiation is that it can be spatially separated (sometimes with just an aperture) from the incident pump beams, and elaborate double monochromators are not required. Because only a very small solid angle is required to collect all of the CARS radiation, interference from background luminosity is small. Furthermore, the CARS beam is in a spectral region (the anti-Stokes region) that is free of laser-induced fluorescence.

Despite all of the significant advantages of CARS, there are a few disadvantages. Although the CARS signal is rather intense, the sensitivity is limited by interferences from the nonresonant background. Polarization-dependent experiments have been used recently to suppress that background but at a cost of losing some of the desired CARS signal. The spatial resolution is not as good as in spontaneous Raman scattering; typical interaction regions are 1 mm long and 0.2 mm in diameter. Another disadvantage is the complex dependence of species concentration and temperature on the CARS signal, requiring extensive analysis and calibration. However, the disadvantages are often far outweighed by the advantages. That is certainly the case in the carbon ablation experiments, where spontaneous Raman methods are precluded because of poor sensitivity and interference from the

[1] G. Adomeit, G. Mohiuddin, and N. Peters, "Boundary Layer Combustion of Carbon," in *Proc. Sixteenth Combustion Symposium*, p. 731 (1977).

[2] K. Matsui, A. Koyama, and K. Uehara, "Fluid-Mechanical Effects on the Combustion Rate of Solid Carbon," *Combust. Flame* 28, 57 (1975).

sample incandescence while CARS has excellent sensitivity with minimal background interference.

A frequency-doubled Nd:YAG laser was used to generate the primary beam (ω_1) at a wavelength of 532 nm. Because frequency doublers are not 100% efficient, there was residual 1064 nm radiation from the laser that was separated from the primary beam by means of a dichroic beamsplitter. The residual 1064 nm beam was further utilized by passing it through a second frequency doubler (Type II KD*P crystal). The primary beam energy was approximately 40 mJ per pulse with a 10^{-8} s pulse width and a 10 Hz repetition rate. The secondary beam energy was about 10 mJ per pulse. We found that the doubling efficiency of the secondary beam could be increased by 20% if the residual 1064 nm beam was passed initially through a quarter-wave plate to improve the polarization. Any 1064 nm radiation remaining after the doubling crystal was absorbed by a Schott KG3 filter oriented at Brewster's angle. The secondary 532 nm beam pumped a dye-laser oscillator, which produced a broadband output centered at ω_2, often referred to as the Stokes beam. The actual frequency was determined by the particular dye and its concentration. The dye-laser output was then increased by passing it through an amplifier that was pumped by 30% of the primary 532 nm beam. The dye-laser beam (ω_2) and the pump beam (ω_1) were adjusted to the same diameter using Galilean telescopes, which ensured optimum focusing of the beams by a 15 cm focal length lens. The beams were aligned so that the optical axis was parallel to the sample surface and the focal region just missed the surface (≤ 200 μm). The incident beams and the newly generated CARS beam were recollimated by a collection lens (15 cm focal length). The CARS beam was separated from the pump beams using apertures, a grating, and a cutoff filter. After passing through a monochromator, the CARS beam was detected by an optical multichannel analyzer (OMA). The OMA output can be stored on floppy disks, and routine data reduction can be performed using an Apple II computer.

2. EXPERIMENTAL APPARATUS

The experimental apparatus is shown schematically in Fig. 1 and in the photographs in Figs. 2 and 3. The ablation apparatus, shown schematically in Fig. 4, was developed by C. H. Hoshall[3] and was modified slightly for use in the optical experiments. The Pyrex chamber was replaced with an aluminum container that had window ports for the laser beams; holes in the base plate were sealed in order to evacuate the chamber, and the vent was placed in the top plate. The carbon sample was electrically heated in an inert atmosphere (either argon or nitrogen), and the sample temperature was measured with a disappearing filament pyrometer. During the ablation experiments, air was metered through a choking orifice and directed perpendicularly to the sample surface to achieve stagnation flow.

The CARS system was modeled after the one developed by Eckbreth at the United Technologies Re-

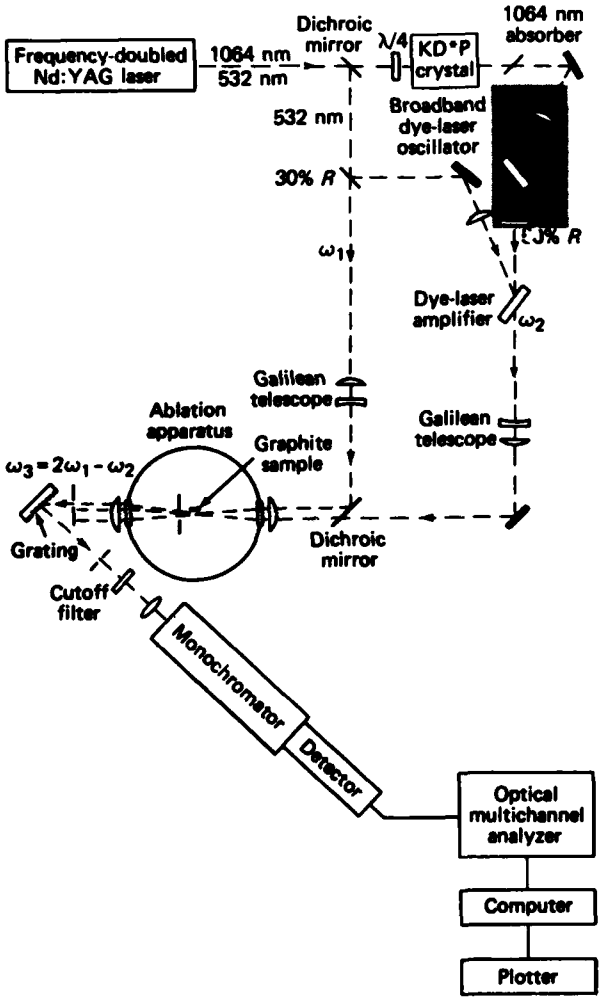

Figure 1 – Schematic of the CARS system and ablation apparatus.

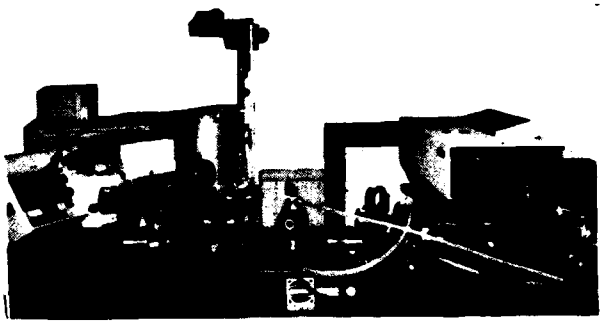

Figure 2 — The CARS system and ablation apparatus.

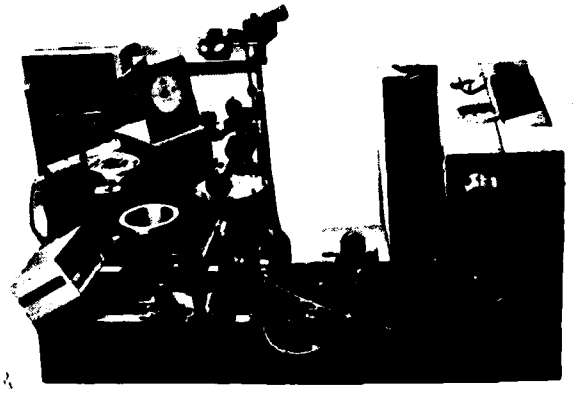

Figure 3 — The CARS system and ablation apparatus with the aluminum shield removed.

[3] R. W. Newman and C. H. Hoshall, *Graphite Ablation in Several Gas Environments*, JHU/APL TG 1336 (Jan 1983).

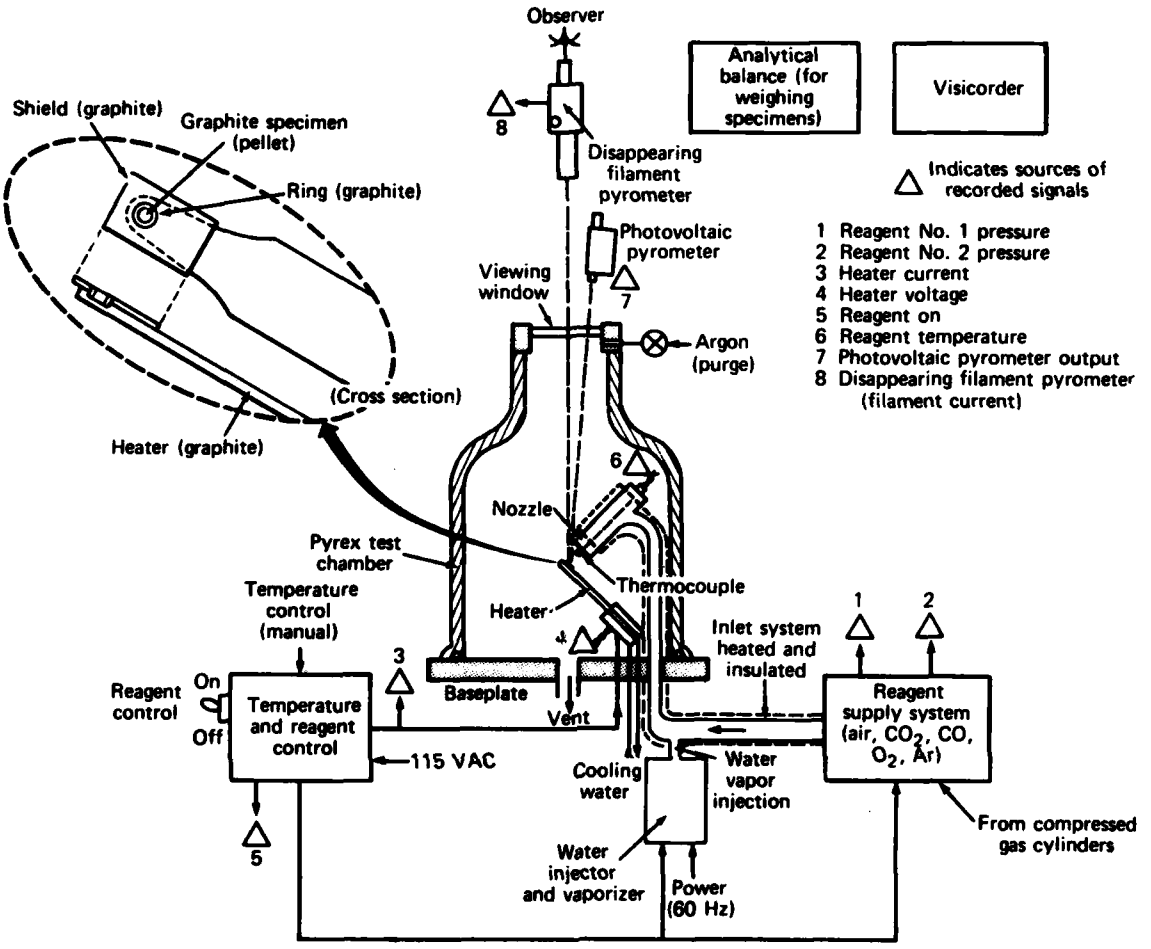

Figure 4 — Schematic of the ablation apparatus prior to modifications for CARS measurements.

search Center.[4,5] Incident pump photons at frequency ω_1 interact with photons at frequency ω_2 through the third-order nonlinear susceptibility of the sample molecules to generate a new beam (the CARS beam) at frequency ω_3, where $\omega_3 = 2\omega_1 - \omega_2$. When the frequency difference $\omega_1 - \omega_2$ is close to the vibrational frequency of a Raman active mode, the magnitude of the generated signal at ω_3 becomes very large. However, the incident beams must be aligned so that the nonlinear interaction is properly phased. In gases, phase-matching can occur when the beams are collinear. Although that is easy to produce optically, the interaction length is rather long, leading to poor spatial resolution. The problem is overcome by separating the incident beams and crossing them at the correct phase-matching angles.

A narrowband laser at a fixed frequency is used to generate ω_1, and because $\omega_1 - \omega_2$ must be varied to coincide with a molecular resonance, ω_2 must be obtained from a tunable source such as a dye laser. There are two methods of tuning ω_2 to generate the CARS spectrum. In one, ω_2 is generated from a narrowband dye laser, and the CARS spectrum is obtained by scanning the dye-laser frequency and using a single-channel detector. In the other method, which is the one we use, ω_2 is generated from a broadband dye laser, and the entire CARS spectrum can be obtained simultaneously for several species using a multichannel detector. Although the latter approach is not as sensitive, it allows fast time-resolved measurements to be made on fluctuating phenomena.

[4] A. C. Eckbreth, "BOXCARS: Crossed-Beam Phase-Matched CARS Generation in Gases," *Appl. Phys. Lett.* 32, 421 (1978).

[5] A. C. Eckbreth and R. J. Hall, "CARS Concentration Sensitivity With and Without Nonresonant Background Suppression," *Combust. Sci. Tech.* 25, 175 (1981).

3. RESULTS

Following the assembly of the optical components and the initial checkout of the system, experiments were conducted on ambient N_2 to gain experience in using this rather complex system. As was discussed earlier, for efficient CARS generation the dye laser frequency, ω_2, must be tuned so that $\omega_1 - \omega_2 = \omega_v$, the frequency of a Raman-active vibrational mode. Because $\omega_v = 2331$ cm^{-1} for N_2, λ_2 must be 607.3 nm. That wavelength was achieved using rhodamine B (Rhodamine 640 from the Exciton Chemical Co.) in the dye laser. As there was no dispersive element in the cavity, the dye laser spectrum was broad (about 200 cm^{-1}), and precise tuning was not necessary. Measurements were obtained with the laser beams aligned collinearly to achieve the maximum CARS signal. Typical CARS signal levels for ambient N_2 were 6×10^5 counts per pulse for a Stokes energy of 8 mJ per pulse. (Neutral density filters were used in front of the OMA to avoid detector saturation.) Measurement of the average power of the CARS beam indicated a conversion efficiency (P_3/P_2) of about 6×10^{-7}, whereas spontaneous Raman efficiencies are typically 10^{-11}. The spatial resolution was improved by crossing the laser beams at a small angle (2 to 3°), causing a slight phase mismatch. Unfortunately, lower sensitivity resulted, but this approach was most expedient in the limited time that was available. (A more complicated optical arrangement that has both good spatial resolution and reasonable sensitivity will be discussed later.)

CO was the next gas to be studied. In that case, $\omega_v = 2143$ cm^{-1} and $\lambda_2 = 600.5$ nm. To shift the dye laser wavelength, the dye concentration was decreased. The resulting dye laser spectrum is shown in Fig. 5. Note that there is still laser output in the N_2 region (607 nm), so that N_2 could also be detected, albeit at a lower sensitivity. Several experiments were also conducted in an evacuable sample cell in which the partial pressures of N_2 and CO could be varied.

The CARS system was then interfaced with the ablation test apparatus. The optical system was aligned so that the optical axis was parallel to the sample surface and the focal region just missed the surface ($\lesssim$200 μm). The sample and heater were made of ATJ graphite and had a combined width of 8.5 mm along the CARS optical axis. For the particular crossing angle we used, the interaction region of the laser beams along the optical axis (i.e., parallel to the carbon surface) was approximately 5 mm. This dimension was determined by passing a small jet of argon perpendicularly through the laser beams and exhausting the jet through a tube that was connected to a mechanical pump. The argon displaced the ambient air, and because argon is not Raman active, a decrease in the N_2 CARS signal was associated with the jet passing through the region where the laser beams overlapped.

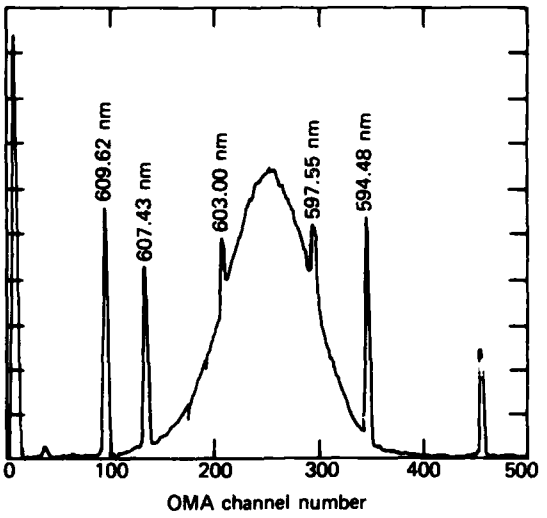

Figure 5 — The dye-laser spectrum. The sharp lines are from a neon calibration lamp. Monochromator: 1200 grooves per mm grating, 75 μm slit width.

The predominant effect of temperature on the CARS signal is to decrease its intensity because the resonant contribution to the signal is proportional to the square of the molecular number density. Thus, the CARS signal should be proportional to T^{-2}. Another effect of temperature is to change the shape of the spectral band. As the temperature increases, the $v = 0 \rightarrow 1$ band broadens as the rotational population distribution shifts to higher energy levels. At higher temperatures, the higher vibrational bands ($v = 1 \rightarrow 2$, $2 \rightarrow 3$, etc.) become visible. Unfortunately, changes in the shape of the CARS bands are not easily discerned by us because of the limited resolution and spectral dispersion of the present monochromator.

The system was calibrated at room temperature using known partial pressures of CO and N_2, and at

≈2000 K using only N_2. (The test chamber was not leaktight; therefore, CO was not calibrated at high temperatures because of the potential reaction with oxygen.) Shown in Fig. 6 is the CARS spectrum of N_2 and CO at room temperature. The partial pressures of N_2 and CO were 610 and 150 Torr, respectively. The N_2 intensity was 8.6×10^4 counts and the CO intensity was 3.8×10^4 counts for an integration time of 3.3 s (33 laser pulses) and a Stokes energy of about 4 mJ per pulse.

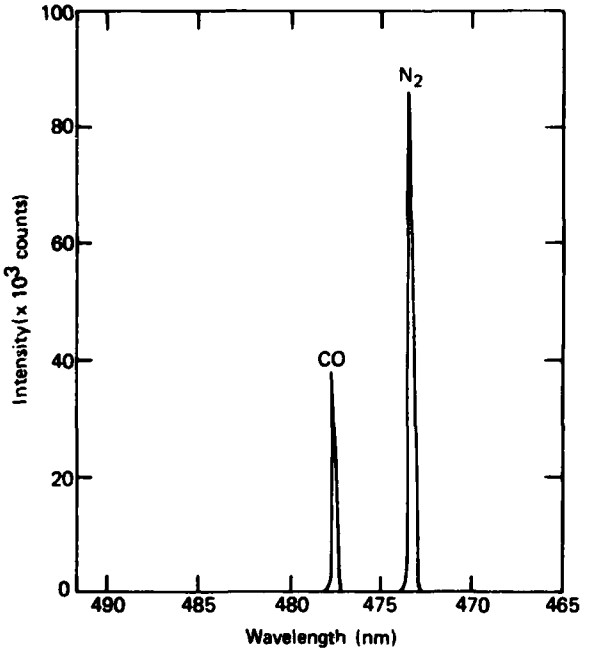

Figure 6 — The CARS spectrum of N_2 and CO at room temperature. P_{CO} = 150 Torr; P_{N_2} = 610 Torr; integration time is 3.3 s (33 laser pulses).

During the initial experiments at 2000 K, the signal-to-noise ratio was limited by the blackbody radiation from the sample and heater. This background interference was minimized, with no decrease in the CARS signal, by installing a graphite aperture near the sample (see Fig. 1). It should be noted that this was possible only because the CARS signal is essentially a small-diameter laser beam and, therefore, only small, solid-angle collection optics are required.

Several ablation runs were made on ATJ graphite heated to about 2000 K with air impinging on the sample at flow velocities ranging from 0.5 to 1.5 m/s. The region probed by the CARS technique was located about 200 μm from the surface. The CARS spectrum is shown in Fig. 7. Although the signal-to-noise ratio was not large, it is evident that CO produced by the oxidation of graphite in air was detected. We found that with a hot sample, the intensity of the N_2 signal depended strongly on the flow velocity of the air. This dependence could have been a result of the gas temperature increasing as the flow velocity decreased. As was discussed earlier, when the gas temperature increases, the band should become asymmetric toward a longer wavelength, and the $v = 1 \rightarrow 2$ band should be apparent. However, that was not observed for N_2. Even with relatively poor spectral resolution, we would have expected to observe changes in the band shape. It is possible that the spatial resolution was worse than we thought, i.e., the interaction length may have extended beyond the heater edge. Because the CARS signal scales as T^{-2},

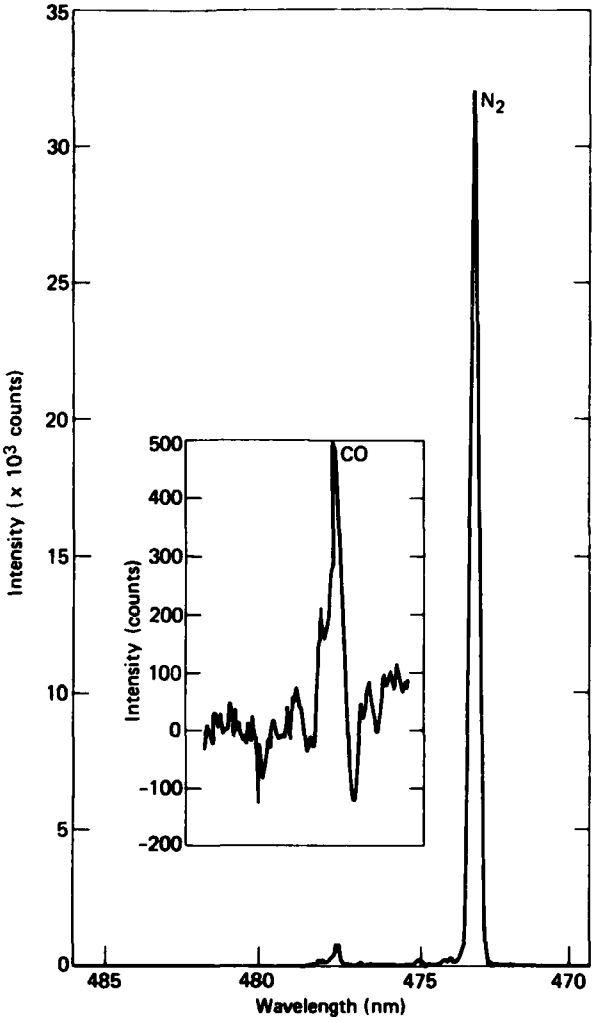

Figure 7 — The CARS spectrum of CO produced during the ablation of ATJ graphite in air. Surface temperature is about 2000 K, airflow velocity is 1 m/s, distance of laser beams from surface is about 200 μm, and integration time is 16.5 s (165 laser pulses).

the gas in the cooler region would dominate the signal.

On the other hand, the measured CO band appeared to be asymmetric, and the $v = 1 \rightarrow 2$ band may have been present. This possibility is consistent with the CO temperature being close to 2000 K, the graphite surface temperature. A possible explanation for the CO temperature appearing higher than that of N_2 is that CO was present only in the hot part of the laser-probed region; by the time the CO had reached the cooler part, it had reacted homogeneously with oxygen to form CO_2. The CO concentration can be estimated using the calibration at room temperature (Fig. 6) and assuming that the gas temperature was 2000 K. If the CARS signal scales as the square of the number density, the observed signal shown in Fig. 7 corresponds to a CO partial pressure of approximately 50 Torr. Actually, as the CO partial pressure decreases to below 100 Torr, the nonresonant contribution from N_2 becomes significant, and the CARS signal no longer depends on the square of the CO number density.[6] Another approach is to determine the CO concentration from the shape of the CARS spectrum.[5,7] On the basis of Hall and Eckbreth's CO spectra,[7] the observed band shape appears to be consistent with CO concentrations in the range of 5 to 10% (40 to 80 Torr).

[6] A. C. Eckbreth and P. W. Schreiber, "Coherent Anti-Stokes Raman Spectroscopy (CARS): Application to Combustion and Gas-Phase Diagnostics," in *Chemical Applications of Nonlinear Spectroscopy*, A. B. Harvey, ed., Academic Press, New York, p. 27 (1981).

[7] R. J. Hall and A. C. Eckbreth, "Combustion Diagnosis by Coherent Anti-Stokes Raman Spectroscopy (CARS)," *Opt. Eng.* **20**, 494 (1981).

4. RECOMMENDATIONS

Although we have demonstrated the feasibility of using CARS to detect the CO produced during the oxidation of graphite, improvements are needed to increase the capability of the experimental technique. The ablation apparatus must be redesigned so that the chamber is leak-tight and evacuable. A micrometer adjustment is needed to move the sample in a direction perpendicular to the laser beams so that measurements can be obtained at precise distances from the graphite surface. Because the CARS signal scales as the square of the interaction length, a larger sample is desirable. The flow field should be characterized to ensure the correct interpretation of the ablation experiments. Matsui et al.[2] used a hot-wire anemometer to characterize the flow field with the sample at room temperature. If optical techniques were used, the flow field would not be perturbed by the diagnostic method, and any effect of the hot sample and/or chemical reactions on the flow could be determined.

In the CARS apparatus, it would be desirable to increase the CARS signal by adding an amplifier to the Nd:YAG laser. The CARS signal depends on the square of the pump laser intensity; therefore, the amplifier would increase the CARS signal by a factor of ten. Better phase matching and spatial resolution should be achieved using a configuration known as BOXCARS.[4] With that technique, the pump beam is split into two components that are crossed at a particular angle with respect to the dye-laser beam. Various angles can be chosen, based on the phase-matching requirements. For gas temperature measurements, extensive modeling of the CARS spectrum is required, and improvements to the monochromator may be necessary. Further work to minimize the blackbody interference is desirable and would require adjustable apertures and, possibly, gating of the OMA. Modifications to the dye laser will improve the amplitude and spectral stability of the output; hence, the CARS signal will be more stable. In the graphite ablation experiments, the simultaneous detection of CO and CO_2 would be desirable but would require additional modifications to the dye laser.

ACKNOWLEDGMENT

We would like to thank A. C. Eckbreth (United Technologies Research Center) for his help in the design of the CARS system and R. Antcliff and O. Jarrett (NASA Langley) for their help in the initial checkout. We also appreciate helpful discussions with C. H. Hoshall, L. W. Hunter, and L. B. Weckesser (APL).

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[1] G. Adomeit, G. Mohiuddin, and N. Peters, "Boundary Layer Combustion of Carbon," in *Proc. Sixteenth Combustion Symposium*, p. 731 (1977).

[2] K. Matsui, A. Koyama, and K. Uehara, "Fluid-Mechanical Effects on the Combustion Rate of Solid Carbon," *Combust. Flame* **25**, 57 (1975).

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